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Design, Synthesis, and SAR Studies of Some Novel Chalcone Derivatives for Potential Insecticidal Bioefficacy Screening on *Spodoptera frugiperda* (Lepidoptera: Noctuidae)

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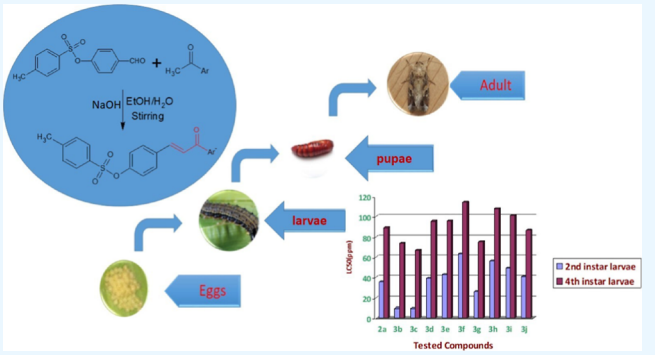
ABSTRACT: *Spodoptera frugiperda* is a species of the order Lepidoptera. It is one of the species of fall armyworm moths distinguished by its larval life stage, is found in different regions of Africa, and can cause incredible damage. This is the first report produced by the preparation of an indexed combinatorial library of novel chalcone derivatives **3a−k** *via* treatment of 4-formylphenyl4-methylbenzenesulfonate (**1**) with some acetyl compounds **2a−k** in the presence of NaOH. The structures of the synthesized compounds were proven by different spectroscopic techniques such as infrared, ^{1}H NMR, ^{13}C NMR, and elemental analyses. In this work, we studied their toxicity effect against *S. frugiperda*, followed by a structure−reaction relationship. Moreover, newly prepared chalcone derivatives were tested as insecticides using *S. frugiperda*. It has been found that most compounds have good to excellent potential effectiveness. Among all of the compounds, **3b**, **3g**, and **3j** exhibited excellent effectiveness. Furthermore, compound **3c** showed the most activity, with LC_{50} = 9.453 ppm of the second instar larva and LC_{50} = 66.930 of the fourth instar larva .



1. INTRODUCTION

Spodoptera frugiperda is one of the most dangerous insects that have recently entered Egypt[1−3] and has affected farmers, particularly those who currently grow maize. It was also responsible for the destruction of the corn crop in the year 2021. It can devour 80 other crops in a few days, including rice, cotton, sugar cane, sorghum, and other important crops.[4,5] Recently, researchers introduced some newly prepared organic compounds, which are sustainable against this insect and harmless to people and the environment.[6] The synthesized compounds that contain a chalcone moiety or a sulfonate group were very effective against *S. frugiperda*,[7] and it is known that chalcones are one of the most important types of natural products belonging to the flavonoid family.[8] Moreover, chalcones are important compounds in medicinal and biological activities such as antileishmanial, antiparasitic,[9] anticancer, anti-inflammatory, cardiovascular, antitumor,[10] and antitubercular activities.[11] Moreover, they represent anti-agents in the agricultural field.[12,13] Accordingly, these compounds have been used in engineered agrochemicals and to formulate manufactured mixtures possessing many pharmacological effects.[14,15] Due to the presence of a conjugated enone system, chalcones are very reactive species, Figure 1.[16] Therefore, chalcone compounds are considered widely applicable for the design and synthesis of novel biological and agricultural fields.[17]

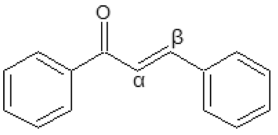


Figure 1. Molecule of chalone.

Therefore, in view of these observations and in conjunction with our previous interest in the synthesis of heterocyclic rings,[18−25] we aimed here to find new insecticides for the synthesis of novel chalcone derivatives and the subsequent assessment of their toxicological and biological bio-efficacy against *S. frugiperda*.

2. RESULTS AND DISCUSSION

2.1. Chemistry. In this paper, it was presented that the reaction between 4-formylphenyl-4-methyl benzenesulfonate (**1**) and acetyl derivatives **2a−k,** such as acetophenone (**2a**), 4-

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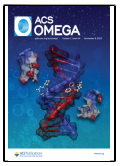

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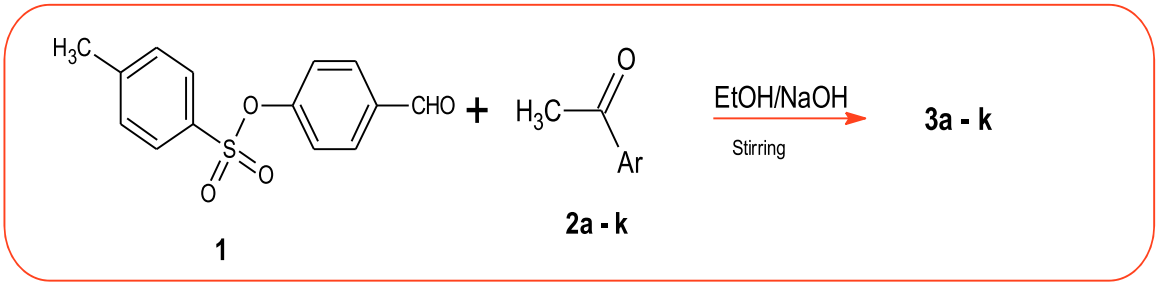
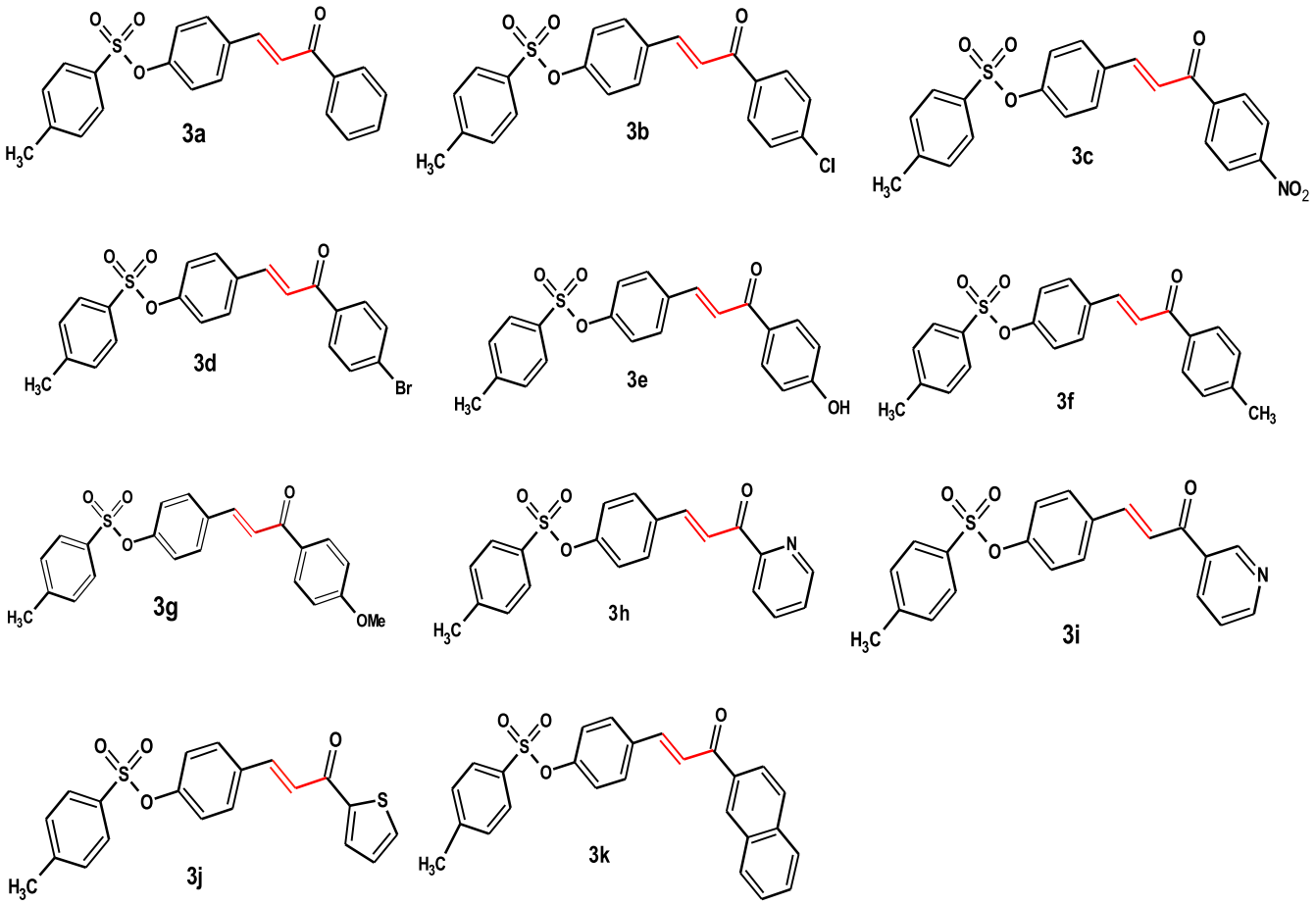
Scheme 1. Synthesis of Chalcone Derivatives





chloroacetophenone (**2b**), 4-bromoacetophenone (**2c**), 4-hydroxyacetophenone (**2d**), 4-methyl-acetophenone (**2e**), 4-methoxyacetophenone (**2f**), 2-acetylpyridine (**2g**), 4-acetylpyridine (**2h**), 2-acetylthiophene (**2i**), and 2-acetylnaphthalene (**2k**), in the presence of alcoholic sodium hydroxide afforded chalcone derivatives **3a**−**k** in excellent yield (Scheme 1). Most of the advanced reactions are safe and easy, and pollution issues associated with toxic solvent use were avoided because we used a green solvent (mixed water and ethanol).

The structures of the obtained chalcones **3a**−**k** were determined by infrared (IR), ^{1}H NMR, ^{13}C NMR, and elemental analyses. The infrared spectra of these synthesized products **3a**−**k** displayed the apparent bands in the region of δ 1678−1655 cm^{-1} due to C═O groups. The ^{1}H NMR (DMSO-d_6) spectra exhibited only aromatic proton signals located in the region δ 8.60−8.25 ppm, in addition to CH_3 groups for all compounds at δ 2.44−2.41 ppm. Moreover, compounds **3e** and **3f** showed new signals at δ 2.40 ppm and at δ 3.88 ppm due to methyl and methoxy groups, respectively. Furthermore, their ^{13}C NMR spectra indicated that new signals appeared at δ 189.62 and at δ 189.00 ppm due to C═O groups. Additionally, elemental analyses and mass spectra confirmed the correct structure.

2.2. Insecticidal Behavior. The laboratory bioassay analysis was conducted to estimate the insecticidal bioefficacy screening of 11 newly synthesized target compounds as toxicity agents against a sensitive strain of the second and fourth larval instar of the *S. frugiperda* pest. The synthesized and tested target compounds after 3 days of treatment gave high to moderate insecticidal activity, with LC_{50} values ranging from 63.100 to 9.543 ppm for second instar larvae and from 114.10 to 66.930 ppm for fourth instar larvae. Thus, the 11 tested target compounds showed insecticidal activity with the above-mentioned data after 3 days of treatment for *S. frugiperda* second and fourth instar larvae. To estimate the toxicological

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Table 1. Insecticidal Efficacy for Recently Synthesized Compounds after 3 Days of the Second and Fourth *S. frugiperdsa* Innstar Larvae

	second instar larvae				fourth instar larvae			
comp.	LC_{50} (ppm)	x^2	slope	toxic ratio	LC_{50} (ppm)	x^2	slope	toxic ratio
3a	35.646	0.887	1.113 ± 0.261	0.265	89.002	0.298	0.954 ± 0.267	0.752
3b	9.616	0.596	0.930 ± 0.259	0.975	73.720	0.232	1.000 ± 0.255	0.907
3c	9.453	0.969	0.711 ± 0.251	1	66.930	0.397	1.061 ± 0.268	1
3d	39.036	0.699	1.040 ± 0.257	0.165	95.720	0.510	0.931 ± 0.288	0.699
3e	42.921	0.377	0.956 ± 0.257	0.150	96.164	0.0958	0.888 ± 0.296	0.695
3f	63.100	0.026	0.776 ± 0.266	0.149	114.10	0.261	0.853 ± 0.287	0.586
3g	26.247	1.538	1.541 ± 0.288	0.360	75.308	1.197	1.003 ± 0.287	0.888
3h	56.260	0.110	0.872 ± 0.255	0.168	107.93	0.186	0.851 ± 0.286	0.620
3i	49.212	0.231	0.928 ± 0.256	0.192	101.36	0.235	0.891 ± 0.287	0.660
3j	40.530	0.533	0.987 ± 0.257	0.232	86.511	0.346	0.968 ± 0.288	0.773
3k	51.232	0.124	0.8625 ± 0.25	0.184	102.32	0.146	0.895 ± 0.277	0.648

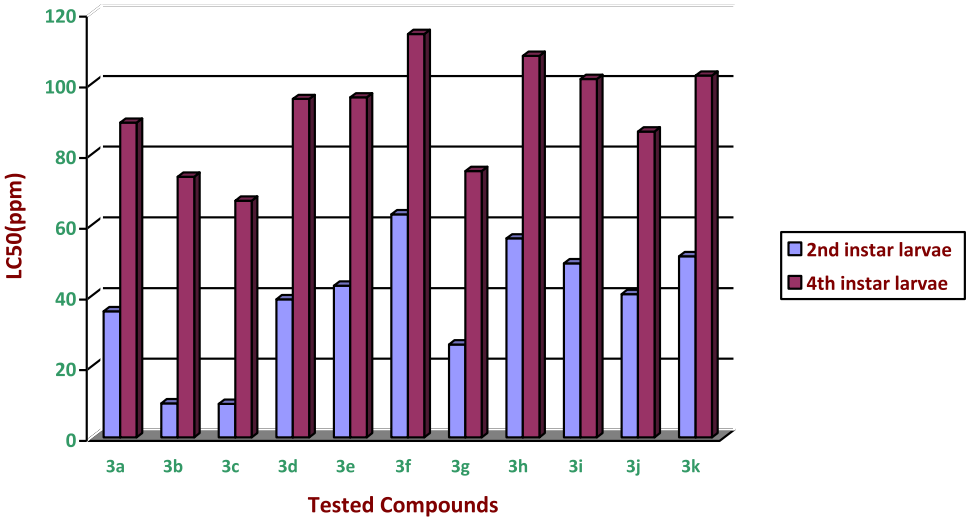

Figure 2. Insecticidal activity of selective chalcone derivatives **3a−k** against the second and fourth *S. frugiperda* instar larvae after treatment.

application of the newly synthesized compounds as insecticidal agents, their toxic activities against *S. frugiperda* were tested. Thus, the LC_{50} values were determined, as shown in Table 1 and Figures 2 and 3. The results show that compound **3c** was more active than other chalcone-synthesized derivatives. The new synthesized compounds were screened for their insecticidal bioefficacy as shown below:

2.2.1. Insecticidal Bioefficacy Screening for the Second Instar Larvae of S. frugiperda after Treatment. As shown in Figures 1 and 2, the results of target compounds **3a−k** tested against the instar larvae after 72 h are shown in Table 1. The results assured that all synthesized compounds have high to low insecticidal activity against the second larvae of *S. frugiperda*, with LC_{50} values ranging from 63.100 to 9.453 ppm, in which the results of compounds **3c**, **3b,** and **3g** have promising insecticidal activity against the second instar larvae of *S. frugiperda*. Compounds **3c**, **3b**, **3g,** and **3a** possess high insecticidal bioefficacy and their LC_{50} values are 9.453, 9.616, 26.24, and 35.64 ppm, in which the toxic ratio is 1, 0.957, 0.360, and 0.265, respectively.

2.2.2. Insecticidal Bioefficacy Screening for the Fourth Instar Larvae of S. frugiperda after Treatment. As shown in Figures 1 and 2, the results of synthesized compounds **3a−k** for the fourth instar larvae are confirmed, and the LC_{50} values are presented in Table 1. All the synthesized compounds exhibit strong to weak insecticidal activity, which showed varied values from 114.100 to 66.930 ppm. The median lethal concentration LC_{50} and slope values of the newly synthesized compounds were computerized by using a regression analysis program and reported as parts per million (ppm), Figure 3. The insecticidal activity of the target compounds **3a−k** had an LC_{50} value as follows: 89.002, 73.720, 66.930, 95.720, 96.164, 114.10, 75.308, 107.93, 101.36, 86.511, and 102.32, respectively, in which second instar larvae are represented by black lines and fourth instar larvae are represented by red lines. As shown in the analysis data, chalcone derivative compound **3c** is more active toward *S. frugiperda* than other synthesized compounds.

3. STRUCTURE−ACTIVITY RELATIONSHIP

In this work, the insecticidal activity was estimated by the authors systematically against second and fourth instar larvae of *S. frugiperda*. In Table 1 and Figures 2 and 3, the high activity associated with compound **3c** may be due to the presence of a nitrogen dioxide (NO_2) moiety and a sulfonate group in the chemical structure, in addition to the conjugated enone system, whose presence in a compound structure makes it a very reactive species. Additionally, compound **3b** gives high activity, which may be attributed to the presence of chloride atoms in their chemical structure group. The insecticidal activity of compounds **3a−k** was compared to the effect of substituents; the

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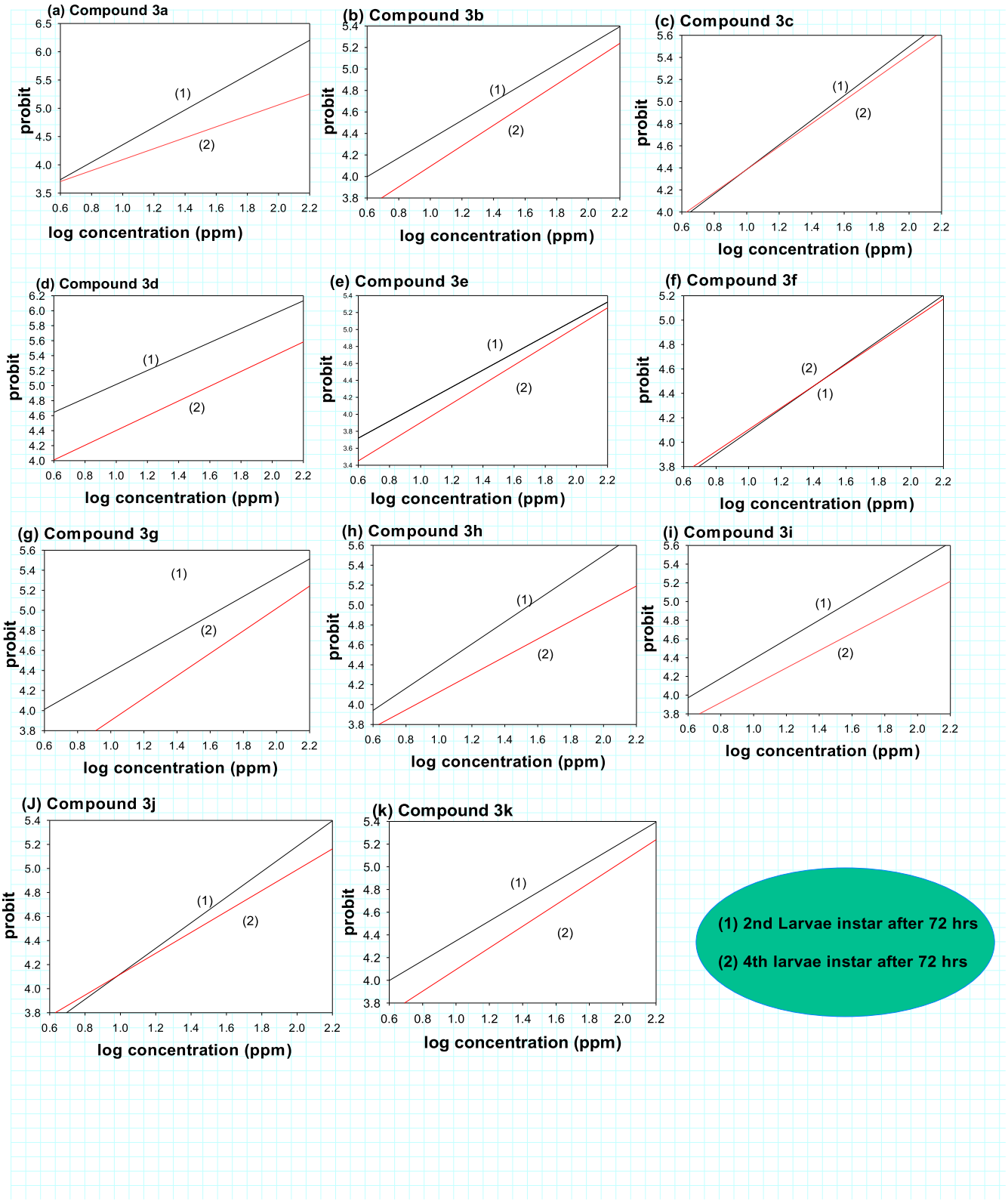

Figure 3. Insecticidal activities of selective compounds **3a−k** for the second and fourth *S. frugiperda* instar larvae after treatment.

obtained results showed that **3c** > **3b** > **3g** > **3j** > **3a** > **3d** > **3f** > **3i** > **3h** > **3k** > **3e**.

4. CONCLUSIONS

By using an environmentally friendly method, we developed and synthesized a novel and ecologically sound series of some new bioactive chalcone derivatives that possess insecticidal toxicity. The prepared novel target compound structures were validated by using IR, ^{1}H, and ^{13}C NMR spectroscopic techniques and elemental analyses. Also, under laboratory conditions, the toxicological evaluation parameters of the newly synthesized products were investigated against *S. frugiperda* and LC_{50} values

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were estimated. Compounds **3c**, **3b**, **3g**, and **3a** show high insecticidal bioefficacy, with LC_{50} values of 9.453, 9.616, 26.247, and 35.646 ppm, respectively.

5. MATERIALS AND METHODS

All the used chemical materials are from Aldrich, and they were used as received without any further purification. By using thin layer chromatography, all reactions were monitored. All melting points were recorded using the Kofler melting point device . IR spectroscopy was measured using a Bruker Alpha platinum-attenuated total reflection spectrophotometer. ^{1}H NMR and ^{13}C NMR data were detected using the Bruker BioSpin AG spectrometer at 400 MHz. The larvae of *S. frugiperda* insects were gathered from the cornfields of the Institute of Plant Protection at Shandweel, Sohag, Egypt.

6. EXPERIMENTAL SECTION

6.1. Chemistry. *6.1.1. Synthesis of Chalcone Derivatives **3a**–**k**.* A sodium hydroxide solution of (1 mmol, 5 mL) was dissolved in water and added drop by drop to a mixture of (1 mmol) compound **1** and acetyl derivatives **2a**–**k**, namely, acetophenone (**2a**), 4-chloroacetophenone (**2b**), 4-bromoacetophenone (**2c**), 4-hydroxyacetophenone (**2d**), 4-methylacetophenone (**2e**), 4-methoxyacetophenone (**2f**), 2-acetylpyridine (**2g**), 4-acetylpyridine (**2h**), 2-acetylthiophene (**2i**), and 2-acetylnaphthalene (**2k**). At room temperature, the mixture was stirred for 5 min in cold, neutralized with dilute HCl solution, washed with water several times, and filtered, and the formed precipitate was crystallized using ethanol.

*6.1.2. 4-[3-Oxo-3-phenylprop-1-en-1-yl]phenyl-4-methylbenzenesulfonate (**3a**).* Yield: 87%; mp 146 °C. IR (ν) (KBr) cm^{-1}: 3050 (C−H aromatic), 2951 (C−H aliphatic), 1661 (C═O), 1331, 1150 (SO_2) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.10−8.17 (m, 13H Ar−H and d, 2H of 2C−H), 2.43 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 188.53, 150.75, 146.43, 145.75, 141.96, 136.20, 134.21, 134.22, 131.83, 130.97, 130.76, 129.40, 128.73, 123.54, 122.47, 21.46; Anal. Calcd for $C_{22}H_{18}O_4S$ (378); calcd C (69.82%), H (4.79%), S (8.47). Found: C (69.81%), H (4.76%), S (8.45%).

*6.1.3. 4-[3-(4-Chlorophenyl)-3-oxoprop-1-en-1-yl]phenyl-4-methylbenzenesulfonate (**3b**).* Yield: 92%; mp 154 °C. IR (ν) (KBr) cm^{-1}: 3069 (C−H aromatic), 2924 (C−H aliphatic), 1662 (C═O), 1369, 1147 (SO_2) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.13−8.29 (m, 12H Ar−H and d, 2H of 2C−H), 2.45 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 188.53, 150.81, 146.43, 143.16, 138.70, 136.56, 134.29, 131.84, 131.05, 130.93, 130.75, 129.38, 128.73, 123.40, 122.97, 21.64; Anal. Calcd for $C_{22}H_{17}ClO_4S$ (412.5); calcd C (64.40%), H (4.15%), S (7.77%). Found: C (64.39%), H (4.16%), S (7.75%).

*6.1.4. 4-[3-(4-Nitrophenyl)-3-oxoprop-1-en-1-yl]phenyl-4-methylbenzenesulfonate (**3c**).* Yield: 88%; mp 138 °C. IR (ν) (KBr) cm^{-1}: 3096 (C−H aromatic), 2935 (C−H aliphatic), 1668 (C═O) 1358, 1141 (SO_2) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 6.72−8.38 (m, 12H Ar−H and d, 2H of 2C−H), 2.45 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 189.87, 151.01, 150.41, 146.46, 144.21, 134.10, 131.91, 131.82, 130.76, 130.38, 128.73, 128.35, 124.32, 123.26, 123.02, 21.64. Anal. Calcd for $C_{22}H_{17}NO_6S$ (423) calcd C (62.40%), H (4.05%), N (3.31%). Found: C (62.38%), H (4.04%), N (3.29%).

*6.1.5. 4-[3-(4-Bromophenyl)-3-oxoprop-1-en-1-yl]phenyl-4-methylbenzenesulfonate (**3d**).* Yield: 83%; mp 159 °C. IR (ν) (KBr) cm^{-1}: 3068 (C−H aromatic), 2986 (C−H aliphatic), 1660 (C═O), 1366, 1140 (SO_2) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 6.72−8.38 (m, 12H Ar−H and d, 2H of 2C−H), 2.45 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 189.62, 150.73, 146.34, 142.70, 137.41, 134.38, 133.70, 131.85, 131.27, 130.97, 129.27, 129.01, 128.73, 123.73, 122.96, 21.64; Anal. Calcd for $C_{22}H_{17}BrO_4S$ (337) calcd C (57.78%), H (3.75%), S (7.01%). Found: C (57.75%), H (3.73%), S (7.00%).

*6.1.6. 4-[3-(4-Hydroxyphenyl)-3-oxoprop-1-en-1-yl]-phenyl-4-methylbenzenesulfonate (**3e**).* Yield: 85%; mp 170 °C. IR (ν) (KBr) cm^{-1}: 3424 (OH), 3057 (C−H aromatic), 2924 (C−H aliphatic), 1655 (C═O), 1369, 1151 (SO_2) ^{1}H NMR (DMSO-d_6): δ 7.02−8.82 (m, 12H Ar−H and d, 2H of 2C−H), 4.72 (s, 1H, OH), 2.45 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 189.62, 151.63, 148.34, 144.70, 138.40, 135.38, 133.70, 132.85, 132.27, 130.97, 128.27, 128.09, 128.63, 124.73, 121.96, 21.64; Anal. Calcd for $C_{22}H_{18}O_5S$ (394) calcd C (66.99%), H (4.60%), S (8.13%). Found: C (66.97%), H (4.58%), S (8.12%).

*6.1.7. 4-[3-(4-Methylphenyl)-3-oxoprop-1-en-1-yl]phenyl 4-methylbenzenesulfonate (**3f**).* Yield: 94%; mp 182 °C. IR (ν) (KBr) cm^{-1}: 3064 (C−H aromatic), 2925 (C−H aliphatic), 1668 (C═O), 1302, 1178 (SO_2) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.09−8.06 (m, 12H Ar−H and d, 2H of 2C−H), 2.43 (s, 3H, CH_3), 2.40 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 189.00, 150.67, 146.40, 144.16, 142.30, 135.41, 134.44, 134.38, 131.84, 130.88, 130.74, 129.82, 129.15, 128.72, 123.73, 122.93, 21.64; Anal. Calcd for $C_{23}H_{20}O_4S$ (392) calcd C (7.39%), H (5.14%), S (8.17%). Found: C (70.39%), H (5.13%), S (8.16%).

*6.1.8. 4-[3-(4-Methoxyphenyl)-3-oxoprop-1-en-1-yl]-phenyl-4-methylbenzenesulfonate (**3g**).* Yield: 92%; mp 144. 163 °C. IR (ν) (KBr) cm^{-1}: 3081 (C−H aromatic), 2968 (C−H aliphatic), 1658 (C═O), 1362, 1151 (SO_2) ^{1}H NMR (DMSO-d_6): δ 7.08−8.16 (m, 12H Ar−H and d, 2H of 2C−H), 3.88 (s, 3H, OCH_3), 2.43 (s, 3H, CH_3); ^{13}C NMR (DMSO-d_6): δ 189.76, 163.82, 150.58, 146.41, 141.92, 134.55, 13.86, 131.43, 130.93, 130.75, 130.03, 128.73, 123.74, 122.92, 114.54, 56.07, 21.64; Anal. Calcd for $C_{23}H_{20}O_5S$ (408) calcd C (67.63%), H (4.94%), S (7.85%). Found: C (67.60%), H (4.91%), S (7.82%).

*6.1.9. 4-[3-Oxo-3-(pyridin-2-yl)prop-1-en-1-yl]phenyl-4-methylbenzenesulfonate(**3h**).* Yield: 70%; mp 142 °C. IR (ν) (KBr) cm^{-1}: 3021 (C−H aromatic), 2918 (C−H aliphatic), 1669 (C═O), 1354, 1146 (SO_2); ^{1}H NMR (DMSO-d_6): δ 6.86−8.68 (m, 12H Ar−H and d, 2H of 2C−H), 2.44 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 194.53, 150.80, 146.44, 143.63, 136.43, 143.11, 132.43, 131.81, 130.94, 130.75, 129.03, 128.51, 128.13, 126.98, 125.37, 122.12, 21.63; Anal. Calcd for $C_{21}H_{17}NO_4S$ (379) calcd C (66.47%), H (4.52%), N (3.69%). Found: C (66.44%), H (4.51%), N (3.67%).

*6.1.10. 4-[3-Oxo-3-(pyridin-2-yl)prop-1-en-1-yl]phenyl-4-methylbenzenesulfonate (**3i**).* Yield: 72%; mp 138 °C. IR (ν) (KBr) cm^{-1}: 3068 (C−H aromatic), 2936 (C−H aliphatic), 1673 (C═O), 1336, 1129 (SO_2); ^{1}H NMR (DMSO-d_6): δ 7.06−8.10 (m, 12H Ar−H and d, 2H of 2C−H), 2.45 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 194.03, 151.80, 145.34, 141.63, 133.43, 133.11, 132.43, 131.81, 130.94, 129.80, 129.03, 128.51, 128.13, 126.98, 126.37, 124.12, 21.63; Anal. Calcd for $C_{21}H_{17}NO_4S$ (379) calcd C (66.47%), H (4.52%), N (3.69%). Found: C (66.44%), H (4.51%), N (3.67%).

*6.1.11. 4-[3-Oxo-3-(thiophen-2-yl)prop-1-en-1-yl]phenyl-4-methylbenzenesulfonate (**3j**).* Yield: 74%; mp 167 °C; IR (ν) (KBr) cm^{-1}: 3083 (C−H aromatic), 2952 (C−H aliphatic), 1667 (C═O), 1335, 1149 (SO_2); ^{1}H NMR (DMSO-d_6): δ 7.12−8.31 (m, 11H Ar−H and d, 2H of 2C−H), 2.44 (s, 3H,

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CH_3). ^{13}C NMR (DMSO-d_6): δ 182.97, 150.57, 146.45, 145.75, 133.43, 133.11, 131.81, 130.94, 129.80, 129.03, 128.51, 128.13, 126.98, 123.54, 122.97, 21.64; Anal. Calcd for $C_{20}H_{16}O_4S_2$ (384) calcd C (62.48%), H (4.19%), S (16.68%). Found: C (62.45%), H (4.16%), S (16.65%).

*6.1.12. 4-[3-(Naphthalen-2-yl)-3-oxoprop-1-en-1-yl] Phenyl-4-methylbenzenesulfonate (**3k**).* Yield: 76%; mp 186 °C. IR (ν) (KBr) cm^{-1}: 3075 (C−H aromatic), 2947 (C−H aliphatic), 1678 (C═O), 1323, 1117 (SO_2); ^1H NMR (DMSO-d_6): δ 7.06−8.10 (m, 12H Ar−H and d, 2H of 2C−H), 2.45 (s, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 193.41, 150.81, 146.41, 143.60, 136.44, 134.13, 133.92, 132.42, 131.84, 130.89, 130.34, 129.02, 128.59, 128.12, 126.96, 125.66, 125.36, 122.99, 21.63; Anal. Calcd for $C_{26}H_{20}NO_4S$ (428) calcd C, (72.88%), H (4.70%), S (7.48%). Found: C, (72.85%), H (4.68%), S (7.45%).

6.2. Biology Assay. *Via* the leaf dipping bioassay method, the toxic activity of all newly prepared chalcone derivatives was established.[12,26−30] For the most active synthesized derivatives, the screening results helped us find out the appropriate concentrations that kill 50% (LC_{50}) of the second and fourth instar larvae of *S. frugiperda*. In this report, for every compound of synthesized chalcone derivatives, we took five concentrations plus 0.1% Tween 80 as the surfactant. We offered similar size to the second and fourth instar larvae and put them in disks (9 cm in diameter) of castor bean leaves. They were then dipped in the prepared concentration for 10 s and left to dry. In glass jars (5 pounds), the larvae were placed, and every treatment was repeated three times (10 larvae per each). The *S. frugiperda* instar larvae were provided by the Shandweel Agricultural Research Station, Sohag, Egypt. The control was prepared as a disk dipped in distilled water and Tween 80, where the untreated larvae were transferred and allowed to feed on castor bean for 48 h. After 72 h, the mortality percentage was recovered for all synthesized compounds. The mortality relapse line measurements were elucidated by probit analysis.[31] Mortality was redressed by Abbott's formula.[32] The harmfulness index was detected by sun equations.[33]

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.2c04814.

IR spectra, ^1H NMR spectra, and ^{13}C NMR spectra (PDF)

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Notes

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